April 27, 1999
Supersedes May 2, 1997

**Iowa Department of Transportation**
PROJECT DEVELOPMENT DIVISION - OFFICE OF MATERIALS
INSTRUCTIONAL MEMORANDUM

Matls. I.M. 364

## METHOD OF TEST
## DETERMINATION OF BITUMEN AND WATER CONTENT
## OF BITUMINOUS SLURRY MIXTURES

### SCOPE

This test method is for determining the bitumen and water contents of bituminous slurry mixtures. The aggregate remaining after extraction may be used for sieve analysis.

### PROCEDURE

A.      Apparatus

1.      Reflux extraction equipment consisting of a metal sample basket with a 425 µm (No. 40) mesh screen bottom, stirring apparatus, condenser, pan for retaining fine aggregate, 4000 ml thermal shock resistant glass beaker, basket assembly holder and hot plate.

2.      Filtering equipment consisting of vacuum extractor, filter paper, vacuum pump as described in I.M. 330, A. Apparatus, Items 1 and 2.

3.      Ventilated oven for drying

4.      Balance with at least 3000 gram capacity and capable of weighing to the nearest 0.5 gram.

5.      Plastic wash bottle

6.      Large pan for mixing and sampling

7.      Large long-handle spoon

8.      Drying pans [4-or 6-liter (4-or 6-quart)] capacity.

9.      Spatula, at least 200 mm (8 in.) in length

10.     Stiff bristle brush, approximately 25 mm (1 in.) wide (such as a close-cropped paint brush)

11.     Stainless steel beaker, 2000 ml low form

12.     Rubber gloves

13.     Eye protection

B.    Reagents

   1.    1-1-1 Trichloroethane

   2.    Methanol, industrial grade

C.    Precautions

   1.    Wear rubber gloves and eye protection when handling solvents.

   2.    Adequate ventilation must be provided.

   3.    Avoid inhalation of vapors.

   4.    Vent the exhaust from the vacuum pump into the exhaust hood or to the outside.

   5.    1-1-1 Trichloroethane in the presence of heat and moisture may form acids that are extremely corrosive to aluminum. Proper precautions must be taken to not allow the solvent to remain in small quantities in the tank of the vacuum extractor.

D.    Sample Preparation

   1.    Place the entire sample, as received, in the large mixing pan.

   2.    Mix the sample with the long-handled spoon, breaking up all lumps, until the sample appears homogeneous. Then immediately transfer 1500 to 1800 grams into the drying pan, which has been previously tared.

E.    Test Procedure

   1.    Immediately after obtaining the sample in Step D2, weigh and record the sample mass (weight) to the nearest 0.5 gram. (E)

   2.    Place the sample in the drying oven. Set at 135 ± 3°C (275 ± 5°F) and dry to a constant mass (weight).

   3.    Allow the sample to cool and weigh to the nearest 0.5 gram. (F)

   4.    Transfer the dry sample to the 2000 ml stainless steel beaker and add about 400 ml of 1-1-1 Trichloroethane.

   5.    Stir the sample with the spatula until the solvent has thoroughly permeated the material.

   6.    Allow the sample to soak for at least 20 minutes.
   7.    Transfer the sample together with the solvent into the metal basket while the basket assembly is held in place in the 4000 ml glass beaker with the basket

assembly holder. Use the wash bottle of solvent and wash all of the sample from the stainless steel beaker into the basket.

8.    Remove the basket assembly holder, attach the basket to the condenser and lower into place in the 4000 ml beaker.

9.    Place the loaded beaker on the hot plate and adjust the heat (medium) so the solvent boils gently.

10.   Continue the refluxing until the solvent dripping from the aggregate is straw-colored (minimum 2 hours). During the refluxing, stir the sample at regular intervals to redistribute the aggregate under the condenser knobs and insure complete exposure of the solvent.

11.   Raise the stirring apparatus, turn the heat off and allow the sample and extraction assembly to cool, then remove from the hot plate.

12.   Remove the basket and fine aggregate pan containing the extracted aggregate from the 4000 ml beaker.

13.   Disassemble the basket from the fine aggregate pan. Transfer the material from the basket into a drying pan and the material from the fine aggregate pan into the beaker of solvent which the assembly was removed from. It may be necessary to use a brush and wash bottle to remove all of the particles of material from the basket and fine aggregate pans.

14.   Place the pan of extracted aggregate in the drying oven at 135 ± 3°C (275 ± 5°F).

15.   Seal the beaker with aluminum foil and allow the filtrate to set undisturbed for at least an hour.

16.   Weigh a vacuum extraction filter paper to the nearest 0.1 gram and position it on the vacuum extractor, which has been placed in the exhaust hood.

17.   Decant the supernatant liquid, which has been allowed to set for at least 1 hour, on the filter. Vacuum away the liquid and wash the aggregate residue with several alternate washings of 1-1-1 Trichloroethane and alcohol until the filtrate is straw-colored.

18.   Remove the filter paper and aggregate residue from the vacuum extractor, place them in the same drying pan that contains the extracted aggregate (Step 12).

19.    Dry the aggregate and filter paper to a constant mass (weight) in an oven maintained at 135 ± 3°C (275 ± 5°F).

20.    Drain the filtrate from the tank of vacuum extractor. (See C5.)

21.    Remove the drying pan from the oven, cool, transfer the contents including the filter paper to a weighing pan previously tared, and weigh to the nearest 0.5 gram. Subtract the mass (weight) of the filter paper (Step 16) and record (G).

22.    Brush the clinging aggregate from the filter paper into the weighing pan and save the extracted aggregate for sieve analysis.

F.    Calculations

1.    Calculate the results as follows:

$$P_A = 100 \ x \ \frac{F - G}{G} \qquad\qquad P_W = 100 \ x \ \frac{E - F}{G}$$

Where:

$P_A$ = Parts bitumen per 100 parts dry Aggregate
$P_W$ = Parts water per 100 parts dry Aggregate
E   = Mass (weight) of sample (wet), grams
F   = Mass (weight) of oven dried sample, grams
G   = Mass (weight) of extracted Aggregate, grams

Example:

Given:   E = 1782.5 grams
         F = 1563.25 grams
         G = 1425.5 grams

$$P_A = 100 \ x \ \frac{1563.25 - 1425.5}{1425.5} = 9.66 \qquad\qquad P_W = 100 \ x \ \frac{1782.5 - 1563.25}{1425.5} = 15.38$$

G.    Report:

Report the bitumen and water results in parts per 100 parts of dry aggregate as follows:

Parts Aggregate  =  100
Parts Bitumen    =   9.7
Parts Water      = 15.4

Note:  Percent bitumen (on dry basis) is calculated as follows:

$$100 \ x \ \frac{P_A}{100 + P_A} = \% \ bitumen$$